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Optimization of Oil and Methanol Ratio, Time, and Reaction Temperature of Biodiesel Production from Kemiri Sunan Oil (*Trisperma reutealis*) with Two Stage Transesterification

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Abstract-*Biodiesel is an alternative fuel which is generally produced from the transesterification process of vegetable oil and methanol, ethanol, or butanol using a catalyst which aims to speed up the reaction. Indonesia, as a tropical country, has the ability to produce vegetable oil, as one of the raw materials used in making biodiesel, in a friendly environment. Vegetable oil can be classified into two, namely edible (food) and non-edible (non-food). The research was conducted using a two-stage transesterification method to determine non-edible vegetable oil. The result showed that biodiesel from kemiri sunan oil can be produced by optimizing the ratio of oil and methanol (1: 4; 1: 5; 1: 6), temperature (55^{0}C, 60^{0}C, 65^{0}C) and reaction time (15 minutes, 30 minutes, 45 minutes), to obtain the highest yield value of 82.79%. In the experiment, analysis of density, viscosity, moisture content and cetane number was also conducted, which also indicated that the viscosity and cetane number obtained did not meet SNI for biodiesel.*

Keywords *- Sunan Candlenut Oil, Biodiesel, Two-Stage Transesterification*

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1. Introduction

Indonesia is a country in Southeast Asia and Oceania, between the Indian and Pacific oceans. It consists of over seventeen thousand islands and is characterized by the tropical climate and abundant natural resources, one of which serves as a raw material for the manufacture of renewable liquid fuel, namely biodiesel [1,2]. It is a form of diesel fuel, generally produced from the transesterification process which occurs between vegetable oil and methanol, ethanol, or butanol in the presence of a catalyst aimed to accelerate the reaction [3,4]. However, one of the raw materials used for the manufacture of biodiesel is renewable vegetable oil, and it aids in producing it on a large scale, in addition, it is also environmentally friendly[5]. Vegetable oil is classified into two types, namely edible oil (food) and non-edible oil (non-food) [6,7,8]. Biodiesel was selected because it serves as a non-edible (non-food) vegetable oil, as well as to breach the gaps between foodstuffs [9].

A particular type of non-edible vegetable oil widely available in Indonesia is kemiri sunan oil. The whitish flesh or pulp and seeds or logs from the candlenut has an oil content of 52%, and 40% respectively [10,11]. Kemiri sunan has high oil content, unique characteristics which enable it to be used for various purposes, its growth is relatively rapid, and it widely develops in the lowlands approximately 1,000 m above sea level. Furthermore, it is exceptionally suitable for a conservation plant [12,13]. It is not widely traded and is an alternative basic material for biodiesel, therefore, it needs to be studied.

Table 1: Characteristics of Sunan Candlenut Oil [14]

No.	Parameter	Value
1	Laurat Acid (C12:0)	0,0473 %
2	Miristat Acid (C14:0)	0,1329 %
3	Palmitat Acid (C16:0)	16,9349 %
4	Palmitoleiat Acid (C16:1)	0,8002 %
5	Stearat Acid (C18:0)	7,6561 %
6	Oleat Acid (C18:1)	38,6318 %
7	Linoleat Acid (C18:2)	35,0184 %
8	Linolenat Acid (C18:3)	0,2026 %
9	Arakidat Acid (C20:0)	0,3227 %
10	Eikosenoat Acid (C20:1)	0,263 %

2. Material And Methods

The raw material used in this research is candlenut, obtained from Tasikmalaya, West Java. The oil realized from it was subjected to preliminary treatment, namely purification by *degumming* using H3PO4 0.8%, followed by neutralization using 0.5N NaOH 1%.

2.1 Esterification

Oil Esterification is carried out by reacting oil with methanol at a ratio of 1:4 (100 mL: 17,13 mL), in the presence of a catalyst H_2SO_4 3 mL at a temperature of 60^{0}C and reaction time of 1 hour.

2.2 Transesterification

Oil Transesterification is carried out in two stages, consequently, in this process, the product of esterification reacts with methanol in the presence of a catalyst KOH [15,16,17]. The first stage of transesterification is based on the ratio 1 mol of oil: 4 mol of methanol (100 mL: 17,13 mL) in the presence of a catalyst KOH which used 1% / w of oil at a temperature and reaction time of 50^{0}C and 30 minutes, respectively. In the second stage of transesterification, differences in variables, such as the mole ratio of oil and methanol (1: 4; 1: 5; 1: 6), temperature (55^{0}C, 60^{0}C, 65^{0}C) and reaction time (15 minutes, 30 minutes, 45 minutes) were detected.

2.3 Biodiesel Analysis

Biodiesel analysis evaluates density, viscosity, moisture content, and yield%. The data from studies carried out on biodiesel were compared with the Indonesian National Standard (SNI).

In this study, the second stage of transesterification used independent variables which were determined using the *Response Surface Methodology (RSM) standard design factor/block/run* : 3/1/6 [18].

In this process, the *summary of variables* includes temperature, namely its minimum, average and maximum values which are 55 ° C, 60 ° C, and 65 ° C respectively. The minimum, average and maximum values for reaction time are 15 minutes, 30 minutes, and 45 minutes respectively. Additionally, the minimum, average and maximum values for the methanol: Oil ratios are 4: 1, 5: 1 and 6: 1 respectively. The results from the differences in variables are shown in Table 2.

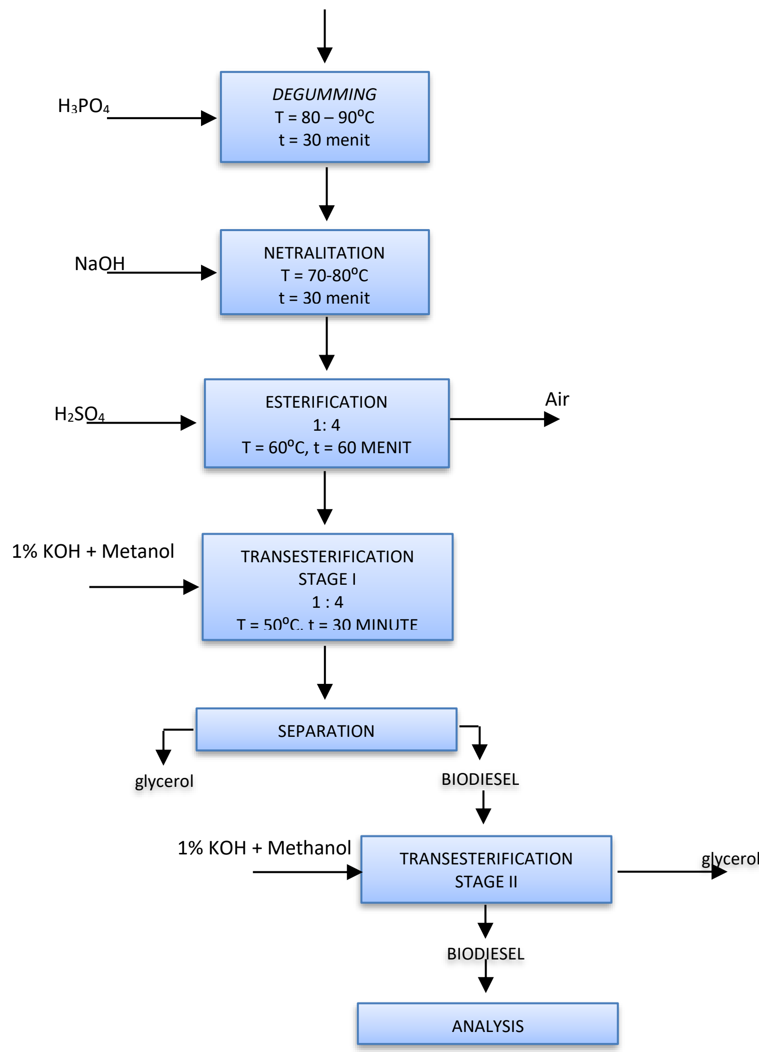


Figure 1. Biodiesel Manufacturing Process

Table 2. Variation in Response Surface Methodoogy Result

Run	A	B	C	Metanol : Oil	Temperature (°C)	Time (Minute)
1	-1,00	-1,00	-1,00	4,00	55,00	15,00
2	-1,00	-1,00	1,00	4,00	55,00	45,00
3	-1,00	1,00	-1,00	4,00	65,00	45,00
4	-1,00	1,00	1,00	6,00	55,00	15,00
5	1,00	-1,00	-1,00	6,00	55,00	45,00
6	1,00	-1,00	1,00	6,00	55,00	15,00
7	1,00	1,00	-1,00	6,00	65,00	45,00
8	1,00	1,00	1,00	3,32	65,00	30,00
9	-1,68	0,00	0,00	6,68	60,00	30,00
10	1,68	0,00	0,00	5,00	51,59	30,00
11	0,00	-1,68	0,00	5,00	68,41	30,00
12	0,00	1,68	0,00	5,00	60,00	4,77
13	0,00	0,00	-1,68	5,00	60,00	55,23
14	0,00	0,00	1,68	4,00	65,00	15,00
15	0,00	0,00	0,00	5,00	60,00	30,00
16	0,00	0,00	0,00	5,00	60,00	30,00

3. Result and Discussion

3.1 Characteristics of Esterification Results of Kemiri Sunan Oil

This stage involves the esterification process which is carried out on the kemiri sunan oil, followed by the characteristic analysis, which is in the form of density, viscosity and free fatty acid (FFA). These values are shown in Table 3.

Table 3: Characteristic Analysis of Kemiri Sunan Oil result of esterification

Characteristic	Unit	Value
Density	kg/m^3	99,5
Viscosity	cP	25,60
FFA	%	6,44

Table 3, shows that during the esterification process there was a conversion of free fatty acids with high molecular weight to methyl ester which has a lower molecular weight, this is due to the decrease in the value of the free fatty acid (FFA) from 25% to 6.44% for kemiri sunan oil. This decrease was caused by the reaction between the free fatty acids in oil and methanol in the presence of an acid catalyst.

3.2 Characteristics of Biodiesel from Kemiri Sunan Oil

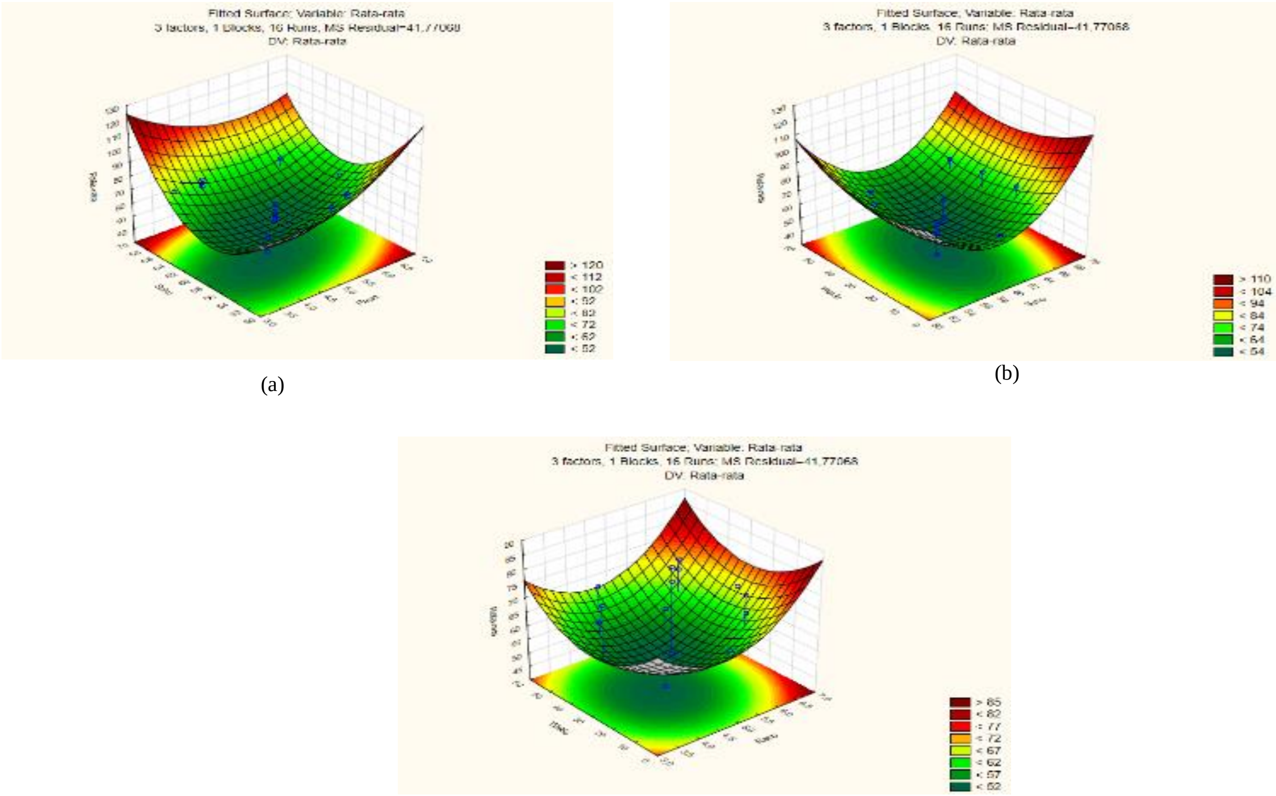
The characteristics of biodiesel derived from kemiri sunan oil were analyzed based on% yield biodiesel, density, viscosity, moisture content, acid number and cetane number.

3.3 Analysis of %Yield Biodiesel

The results obtained are shown in Table 4.

Table 4: %Yield biodiesel

RUN	%Yield Biodiesel (I)	%Yield Biodiesel (II)	%Yield Biodiesel
1	52,29	52,28	52,29 ± 0,007
2	62,36	62,35	62,35 ± 0,007
3	75,00	74,81	74,91 ± 0,133
4	73,73	74,08	73,90 ± 0,250
5	71,67	71,72	71,70 ± 0,036
6	67,74	67,76	67,75 ± 0,015
7	75,27	75,29	75,28 ± 0,015
8	77,95	77,88	77,91 ± 0,052
9	67,99	67,95	67,97 ± 0,029
10	82,79	82,80	82,79 ± 0,007
11	77,70	77,68	77,69 ± 0,015
12	64,03	63,93	63,98 ± 0,074
13	58,60	58,59	58,59 ± 0,007
14	79,47	79,42	79,45 ± 0,037
15	52,51	52,47	52,49 ± 0,029
16	52,47	52,45	52,46 ± 0,015



(a) (b) (c)

Figure 2: Response fitted surface variable

Based on the table, the ideal% yield results are obtained in the 10th variable, with a mole ratio of 1: 5, the temperature of 51.59^{0}C and reaction time of 30 minutes. The biodiesel data from the yield analysis (Table 3.) is processed using the *response fitted surface methodology* (RSM).

In Figure 2 (a) shows that the optimum value is achieved at a yield of 60 to 70%, the temperature of approximately 66 to 68^{o}C and a ratio of 5 mol of methanol: 1 mol of kemiri sunan oil. Figure 2 (b) shows that the optimum value obtained is 66^{o}C within a reaction time of relatively 40 to 50 minutes, and a % yield of approximately 50 to 60%. Figure 2 (c) shows that the optimum ratio of methanol: oil is approximately 5 to 5.5: 1 at an optimum reaction time of 40 to 50 minutes as well as a % yield value of relatively 60 to 70%. Therefore, it was concluded that the optimum conditions in this process occurred at a ratio of 5: 1, temperature of approximately 66 to 68^{0}C and reaction time of 40 to 50 minutes. The processed data was obtained at a determination coefficient of 83.89% (R^2 = 0.83892).

Figure 3 is a Pareto diagram, which shows the most influential variable in this study, namely temperature. The comparative data obtained from the experiments carried out, including estimates from the RSM are shown in Figure 4.

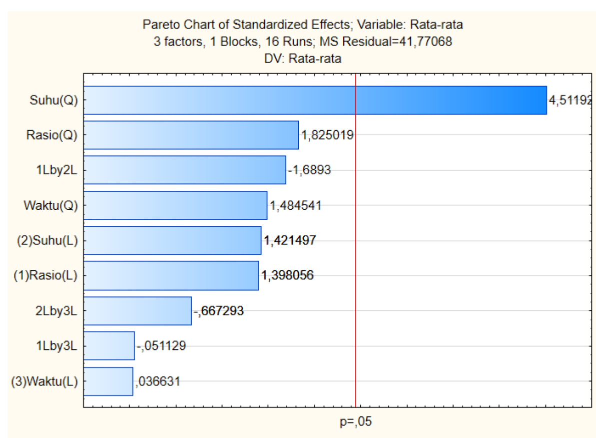


Figure 3: Pareto Diagram of Research Results

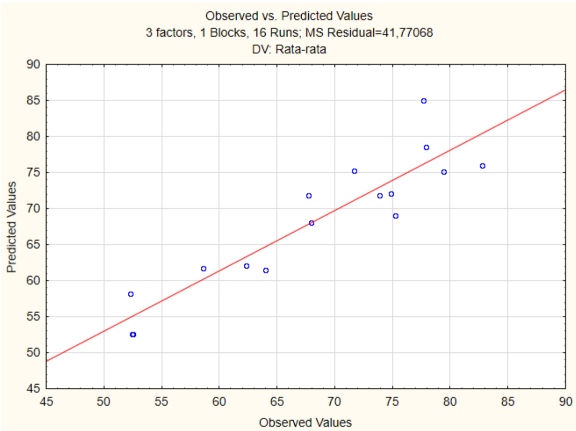


Figure 4. Comparison of Experimental Results and Prediction

3.4 Characteristics of Kemiri Sunan Oil

The results from the experiment carried out on kemiri sunan oil were compared to the existing SNI for biodiesel. The comparison between the experimental biodiesel and SNI Biodiesel 7182-2015 is shown in table 5.

Table 5: Comparison of the Characteristics of Biodiesel from Experimentation with SNI

Parameter	Biodiesel Experimentation	SNI Biodiesel 7182-2015
Density at 40^0 C (kg/m^3)	858 – 906	850 – 890
Setan Number (min) -B100	19,6	51
(Blanco) Airy Shaw (%volume, maks)	0,01 – 0,06	0,05
Viskosity at 40^0C (mm^2/s)	6,78 – 6,99	2,3 – 6,0

It is evident that the average density of kemiri sunan oil meets the standards, as well as the water content analysis. However, the analysis of demon numbers and viscosity of the experimental biodiesel produced is inconsistent SNI 7182-2015.

4.Conclusion

The extraction of biodiesel from kemiri sunan oil is carried out in several phases, namely degumming, esterification, and two-stage transesterification, which was carried out twice because candlenut oil has high *fat fatty acid* (FFA), approximately 15.25%, and is still relatively large 6.44%, even after esterification. The maximum% yield obtained was 82.79%. The optimum conditions according to the graph of *Response Fitted Surface* are the mole ratio of kemiri sunan oil to methanol, temperature and reaction time which are 1: 5, 66 to 68^0C, and 40 to 50 minutes with a determination coefficient of 83, 89% (R^2 = 0.83892).

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